The Crystal Structure of 3a,5,6a-Triphenyl-3,3a-dihydro-2*H*-furo[3,2-*b*]pyrrole-2,6(6a*H*)-dione

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Synopsis. The structure of 3a,5,6a-triphenyl-3,3a-dihydro-2*H*-furo[3,2-*b*]pyrrole-2,6(6a*H*)-dione in the crystalline state was determined by X-ray analysis. The crystals are monoclinic, space group P2/a, a=16.606(8), b=7.314(2), c=16.295(9) Å, β=111.67(4), z=4. The final R value was 0.067 for 2453 reflections.

Recently, we reported the formation of 3,3a-dihydro-2*H*-furo[3,2-*b*]pyrrole-2,6(6a*H*)-diones by the air-oxidation of the enamines which were formed by the condensation of 1,3-diketones with 3-aminopropanates.[1] In the IR spectra of this novel heterocycle, two carbonyl bands were characteristically observed at 1800—1790 and 1760—1755 cm^{-1}, respectively, measured as a KBr pellet. Of these, the one at the higher wave number is reasonably attributable to the carbonyl group of the cyclic ester, while the other seems unusual for the 1-pyrrolin-3-one carbonyl which is formally conjugated with the C=N bond.

In this context, the crystal structure of the titled compound (**1**) has been determined by X-ray analysis.

Results and Discussion

Crystal Data. $C_{24}H_{17}NO_3$, MW=367.4, monoclinic, space group P2/a, a=16.606(8), b=7.314(2), c=16.295(9) Å, β=111.67(4)°, Z=4, D_x=1.33 g cm^{-3}, μ(Cu K_α=1.5418 Å)=0.67 mm^{-1}. The intensities of 2453 observable reflections with $F_o \geq 2\sigma F_0$ ($2\theta \leq 128°$) were collected on Rigaku AFC5 apparatus equipped with a rotating-anode X-ray generator and using a graphite monochromator. The structure was solved by direct methods using MULTAN[2] and refined by block-diagonal least-squares methods. All the H atoms were located in a difference Fourier map. Final cycles of least squares (non-hydrogen atoms anisotropic, H atoms isotropic) gave R=0.067 (R_w=0.071, w=1.0). The positional parameters for non-hydrogen atoms are listed in Table 1.[3]

A perspective drawing of the molecular structure is shown in Fig. 1, and the bond lengths and angles are listed in Tables 2 and 3, which are within usual values. The two carbonyl groups have almost the same bond lengths. The 1-pyrroline ring is planar within ±0.080(19) Å; the oxygen atom of the carbonyl group deviates by 0.254(19) Å from the plane and the torsion angle O(3)–C(5)–C(6)–N(1) is calculated to be 170.3(4)°. From the above results and the C(5)–C(6) bond length of 1.506(5) Å, it is suggested that there is less delocalization of electrons at the O=C–C=N moiety. The tetrahydrofuran ring is close to envelope conformation,

Table 1. Fractional atomic coordinates (×10⁴) and thermal parameters with estimated standard deviations in parentheses

Atom	x	y	z	B_{eq}/Å²
O(1)	3151(2)	1758(4)	6176(2)	2.38(7)
O(2)	3522(2)	4191(5)	5575(2)	3.60(9)
O(3)	3804(2)	−1173(4)	7586(2)	3.48(9)
N(1)	3577(2)	3363(4)	8071(2)	2.08(8)
C(1)	3191(2)	3616(6)	6062(2)	2.43(11)
C(2)	2774(3)	4613(6)	6609(3)	2.38(11)
C(3)	2747(2)	3192(5)	7292(2)	1.75(9)
C(4)	2808(2)	1311(5)	6857(2)	1.80(10)
C(5)	3591(2)	404(5)	7561(2)	2.15(10)
C(6)	4029(2)	1896(5)	8207(2)	2.01(10)
C(7)	1982(2)	3478(5)	7579(2)	2.06(10)
C(8)	2113(3)	3921(6)	8450(3)	3.15(12)
C(9)	1397(3)	4256(7)	8690(3)	4.49(16)
C(10)	569(3)	4104(7)	8081(3)	4.22(16)
C(11)	432(3)	3672(6)	7215(3)	3.64(14)
C(12)	1139(3)	3386(6)	6960(3)	2.72(12)
C(13)	2034(2)	73(5)	6500(2)	1.86(10)
C(14)	1631(3)	−261(7)	5609(3)	3.04(12)
C(15)	914(3)	−1438(8)	5315(3)	4.39(16)
C(16)	608(3)	−2245(7)	5909(4)	4.26(16)
C(17)	1014(3)	−1911(6)	6797(3)	3.82(14)
C(18)	1731(3)	−764(6)	7099(3)	2.85(12)
C(19)	4876(2)	1696(5)	8927(2)	2.02(10)
C(20)	5501(3)	532(6)	8847(3)	2.85(12)
C(21)	6311(3)	415(7)	9528(3)	3.41(13)
C(22)	6486(3)	1461(7)	10278(3)	3.39(13)
C(23)	5868(3)	2594(7)	10375(3)	3.08(12)
C(24)	5058(3)	2716(6)	9696(3)	2.56(11)

Table 2. Bond lengths (l/Å) with estimated standard deviations in parentheses

Bond distance		l/Å	Bond distance		l/Å
O(1)	–C(1)	1.377(5)	C(8)	–C(9)	1.405(5)
O(1)	–C(4)	1.459(4)	C(9)	–C(10)	1.371(6)
O(2)	–C(1)	1.196(4)	C(10)	–C(11)	1.382(7)
O(3)	–C(5)	1.203(5)	C(11)	–C(12)	1.398(4)
N(1)	–C(3)	1.494(4)	C(13)	–C(14)	1.378(5)
N(1)	–C(6)	1.281(5)	C(13)	–C(18)	1.394(5)
C(1)	–C(2)	1.504(5)	C(14)	–C(15)	1.402(6)
C(2)	–C(3)	1.536(5)	C(15)	–C(16)	1.382(6)
C(3)	–C(4)	1.568(5)	C(16)	–C(17)	1.374(7)
C(3)	–C(7)	1.521(4)	C(17)	–C(18)	1.389(5)
C(4)	–C(5)	1.532(5)	C(19)	–C(20)	1.386(5)
C(4)	–C(13)	1.502(4)	C(19)	–C(24)	1.392(5)
C(5)	–C(6)	1.505(5)	C(20)	–C(21)	1.395(5)
C(6)	–C(19)	1.468(4)	C(21)	–C(22)	1.378(7)
C(7)	–C(8)	1.393(5)	C(22)	–C(23)	1.373(5)
C(7)	–C(12)	1.393(5)	C(23)	–C(24)	1.394(5)

TABLE 3. BOND ANGLES (ϕ/°) WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

Bond angle			ϕ/°	Bond angle			ϕ/°
C(1)	-O(1)	-C(4)	112.1(2)	C(3)	-C(7)	-C(12)	120.1(2)
C(3)	-N(1)	-C(6)	112.0(3)	C(3)	-C(7)	-C(8)	120.7(2)
O(1)	-C(1)	-C(2)	110.0(2)	C(8)	-C(7)	-C(12)	119.1(2)
O(2)	-C(1)	-C(2)	130.4(8)	C(7)	-C(8)	-C(9)	119.6(3)
O(1)	-C(1)	-O(2)	119.6(5)	C(8)	-C(9)	-C(10)	120.7(4)
C(1)	-C(2)	-C(3)	104.2(3)	C(9)	-C(10)	-C(11)	120.0(2)
N(1)	-C(3)	-C(2)	107.2(2)	C(10)	-C(11)	-C(12)	119.9(3)
N(1)	-C(3)	-C(4)	105.4(2)	C(7)	-C(12)	-C(11)	120.5(3)
C(2)	-C(3)	-C(4)	104.0(1)	C(4)	-C(13)	-C(18)	118.2(2)
C(2)	-C(3)	-C(7)	112.4(3)	C(4)	-C(13)	-C(14)	121.9(3)
N(1)	-C(3)	-C(7)	109.9(2)	C(14)	-C(13)	-C(18)	119.9(4)
C(4)	-C(3)	-C(7)	117.4(3)	C(13)	-C(14)	-C(15)	119.3(3)
O(1)	-C(4)	-C(13)	111.1(2)	C(14)	-C(15)	-C(16)	120.5(4)
O(1)	-C(4)	-C(3)	104.8(3)	C(15)	-C(16)	-C(17)	119.9(5)
O(1)	-C(4)	-C(5)	101.8(1)	C(16)	-C(17)	-C(18)	120.2(4)
C(3)	-C(4)	-C(5)	102.7(2)	C(13)	-C(18)	-C(17)	120.1(3)
C(3)	-C(4)	-C(13)	121.0(2)	C(6)	-C(19)	-C(20)	121.0(3)
C(5)	-C(4)	-C(13)	113.4(4)	C(6)	-C(19)	-C(24)	119.4(3)
O(3)	-C(5)	-C(4)	126.7(4)	C(20)	-C(19)	-C(24)	119.5(2)
O(3)	-C(5)	-C(6)	127.7(3)	C(19)	-C(20)	-C(21)	119.9(4)
C(4)	-C(5)	-C(6)	105.5(3)	C(20)	-C(21)	-C(22)	119.8(4)
N(1)	-C(6)	-C(19)	123.3(5)	C(21)	-C(22)	-C(23)	121.0(3)
N(1)	-C(6)	-C(5)	112.7(2)	C(22)	-C(23)	-C(24)	119.4(4)
C(5)	-C(6)	-C(19)	124.0(5)	C(19)	-C(24)	-C(23)	120.3(4)

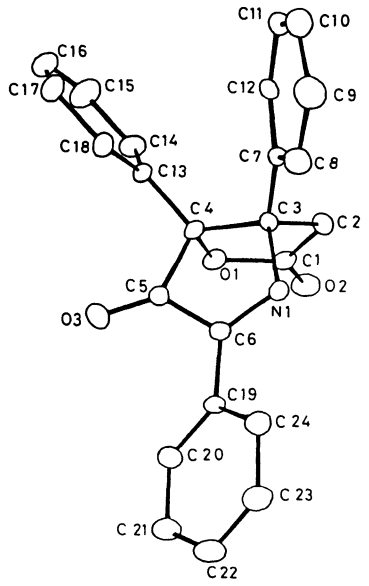

Fig. 1. A perspective drawing of **1**.

C(3) being 0.333(16)Å out of the least-squares plane formed by the other four atoms. The two heterocyclic rings are cis-fused to each other, and the dihedral angle of the rings is 104.1(2)°. The dihedral angle of the phenyl group on C(3) and C(4) is 45.1(2)°. The crystal structure is stabilized mainly by van der Waals forces; the shortest intermolecular distance is 3.128(4) A for O(2)···C(1) (1/2−x, y, 1−z).

References

1) S. Mataka, K. Uehara, K. Takahashi, M. Tashiro, K. Yoshihira, K. Kawazoe, S. Sato, and C. Tamura, *Synthesis*, **1984**, 663.

2) G. Germain, P. Main, and M. M. Woolfson, *Acta Cryst.*, **A27**, 368 (1971).

3) Thermal parameters for the non-hydrogen atoms, fractional coordinates and isotropic thermal parameters for hydrogen atoms, bond lengths involving hydrogen atoms, and observed and calculated structure factors are kept at the Chemical Society of Japan (Document No. 8536).